
**Nickels, ferronickels and nickel
alloys — Standards for the
determination of chemical
composition**

*Nickels, ferronickels et alliages de nickel — Normes pour la
détermination de la composition chimique*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Nickels, ferronickels and nickel alloys — Standards for the determination of chemical composition

1 Scope

This document lists the International Standards which are currently available for the determination of the chemical composition of nickels, ferronickels and nickel alloys (see [Clause 4](#)).

It provides details on the range of application and gives the principle of the method described in each International Standard (see [Clause 5](#)).

[Annex A](#) shows graphical representations of the content ranges of the methods listed in this document:

- [Figure A.1](#) represents the fields of application of the methods available for the three kinds of matrixes;
- [Figure A.2](#) gives the content ranges of the methods for nickels;
- [Figure A.3](#) gives the content ranges of the methods for ferronickels;
- [Figure A.4](#) gives the content ranges of the methods for nickel alloys.

[Annex B](#) provides a bilingual key of the abbreviated terms used in the figures given in [Annex A](#).

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

referee method

stoichiometric method or a method calibrated against pure metals or stoichiometric compounds, which is used for certification analysis or in case of arbitration

Note 1 to entry: Due to a lack of accuracy or to a low number of laboratories having participated in the related validation tests, some stoichiometric methods or methods calibrated against pure metals or stoichiometric compounds cannot be taken as “referee methods”. They are published as Technical Specifications or Technical Reports.

[SOURCE: CEN/TR 10261:2018, modified — Note 1 to entry has been added.]

3.2

routine method

method calibrated against reference materials, certified reference materials or against standard solutions commercially available that is widely used for control purposes (day-to-day analysis)

Note 1 to entry: By agreement, routine methods can be used for any commercial and/or arbitration purposes.

[SOURCE: ISO/TR 9769:2018, modified — Note 1 to entry has been added.]

3.3

stoichiometry

founded on the “law of conservation of mass” where the total mass of the reactants equals the total mass of the products, leading to the insight that the relations among quantities of reactants and products typically form a ratio of positive integers

4 International Standards for the determination of the chemical composition of nickels, ferronickels and nickel alloys

4.1 Nickels

4.1.1 Silver, Ag (referee methods)

ISO 6351:1985, *Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method*

ISO 7523:1985, *Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method*

4.1.2 Arsenic, As (referee method)

ISO 7523:1985, *Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method*

4.1.3 Boron, B (referee method)

ISO 11436:1993, *Nickel and nickel alloys — Determination of total boron content — Curcumin molecular absorption spectrometric method*

4.1.4 Bismuth, Bi (referee methods)

ISO 6351:1985, *Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method*

ISO 7523:1985, *Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method*

4.1.5 Cadmium, Cd (referee methods)

ISO 6351:1985, *Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method*

ISO 7523:1985, *Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method*

4.1.6 Cobalt, Co (referee method)

ISO 6351:1985, *Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method*

4.1.7 Copper, Cu (referee method)

ISO 6351:1985, *Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method*

4.1.8 Iron, Fe (referee method)

ISO 6351:1985, *Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method*

4.1.9 Manganese, Mn (referee method)

ISO 6351:1985, *Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method*

4.1.10 Phosphorus, P (referee method)

ISO 11400:1992, *Nickel, ferronickel and nickel alloys — Determination of phosphorus content — Phosphovanadomolybdate molecular absorption spectrometric method*

4.1.11 Lead, Pb (referee methods)

ISO 6351:1985, *Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method*

ISO 7523:1985, *Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method*

4.1.12 Antimony, Sb (referee method)

ISO 7523:1985, *Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method*

4.1.13 Sulfur, S (referee method)

ISO 7527:1985, *Nickel, ferronickel and nickel alloys — Determination of sulfur content — Iodimetric titration method after induction furnace combustion*

4.1.14 Selenium, Se (referee method)

ISO 7523:1985, *Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method*

4.1.15 Tin, Sn (referee method)

ISO 7523:1985, *Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method*

4.1.16 Tellurium, Te (referee method)

ISO 7523:1985, *Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method*

4.1.17 Thallium, Tl (referee method)

ISO 7523:1985, *Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method*

4.1.18 Zinc, Zn (referee method)

ISO 6351:1985, *Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method*

4.2 Ferronickels

4.2.1 Carbon, C (routine method)

ISO 7524:2020, *Ferronickels — Determination of carbon content — Infrared absorption method after induction furnace combustion*

4.2.2 Chromium, Cr (referee method)

ISO 23156:2021, *Ferronickels — Determination of phosphorus, manganese, chromium, copper and cobalt contents — Inductively coupled plasma optical emission spectrometric method*

4.2.3 Cobalt, Co (referee method)

ISO 7520:1985, *Ferronickel — Determination of cobalt content — Flame atomic absorption spectrometric method*

4.2.4 Cobalt, Co (referee method)

ISO 23156:2021, *Ferronickels — Determination of phosphorus, manganese, chromium, copper and cobalt contents — Inductively coupled plasma optical emission spectrometric method*

4.2.5 Copper, Cr (referee method)

ISO 23156:2021, *Ferronickels — Determination of phosphorus, manganese, chromium, copper and cobalt contents — Inductively coupled plasma optical emission spectrometric method*

4.2.6 Manganese, Mn (referee method)

ISO 23156:2021, *Ferronickels — Determination of phosphorus, manganese, chromium, copper and cobalt contents — Inductively coupled plasma optical emission spectrometric method*

4.2.7 Nickel, Ni (referee method)

ISO 6352:1985, *Ferronickel — Determination of nickel content — Dimethylglyoxime gravimetric method*

4.2.8 Phosphorus, P (referee method)

ISO 11400:1992, *Nickel, ferronickel and nickel alloys — Determination of phosphorus content — Phosphovanadomolybdate molecular absorption spectrometric method*

4.2.9 Phosphorus, P (referee method)

ISO 23156:2021, *Ferronickels — Determination of phosphorus, manganese, chromium, copper and cobalt contents — Inductively coupled plasma optical emission spectrometric method*

4.2.10 Sulfur, S (routine method)

ISO 7526:2020, *Ferronickels — Determination of sulfur content — Infrared absorption method after induction furnace combustion*

4.2.11 Sulfur, S (referee method)

ISO 7527:1985, *Nickel, ferronickel and nickel alloys — Determination of sulfur content — Iodimetric titration method after induction furnace combustion*

4.2.12 Silicon, Si (referee method)

ISO 8343:1985, *Ferronickel — Determination of silicon content — Gravimetric method*

4.3 Nickel alloys**4.3.1 Aluminium, Al (referee method)**

ISO 7530-7:1992, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 7: Determination of aluminium content*

4.3.2 Boron, B (referee method)

ISO 11436:1993, *Nickel and nickel alloys — Determination of total boron content — Curcumin molecular absorption spectrometric method*

4.3.3 Cobalt, Co (referee method)

ISO 7530-1:2015, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 1: Determination of cobalt, chromium, copper, iron and manganese*

4.3.4 Chromium, Cr (referee methods)

ISO 7529:2017, *Nickel alloys — Determination of chromium content — Potentiometric titration method with ammonium iron(II) sulfate*

ISO 7530-1:2015, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 1: Determination of cobalt, chromium, copper, iron and manganese*

4.3.5 Copper, Cu (referee method)

ISO 7530-1:2015, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 1: Determination of cobalt, chromium, copper, iron and manganese*

4.3.6 Iron, Fe (referee method)

ISO 7530-1:2015, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 1: Determination of cobalt, chromium, copper, iron and manganese*

4.3.7 Manganese, Mn (referee method)

ISO 7530-1:2015, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 1: Determination of cobalt, chromium, copper, iron and manganese*

4.3.8 Molybdenum, Mo (referee method)

ISO 11435:2011, *Nickel alloys — Determination of molybdenum content — Inductively coupled plasma atomic emission spectrometric method*

4.3.9 Niobium, Nb (referee method)

ISO 22033:2011, *Nickel alloys — Determination of niobium — Inductively coupled plasma atomic emission spectrometric method*

4.3.10 Nickel, Ni (referee method)

ISO/TS 18223:2015, *Nickel alloys — Determination of Nickel content — Inductively coupled plasma atomic emission spectrometric method*

4.3.11 Phosphorus, P (referee methods)

ISO 9388:1992, *Nickel alloys — Determination of phosphorus content — Molybdenum blue molecular absorption spectrometric method*

ISO 11400:1992, *Nickel, ferronickel and nickel alloys — Determination of phosphorus content — Phosphovanadomolybdate molecular absorption spectrometric method*

4.3.12 Lead, Pb (referee method)

ISO 11437:2018, *Nickel alloys — Determination of lead — Electrothermal atomic absorption spectrometric method*

4.3.13 Sulfur, S (referee method)

ISO 7527:1985, *Nickel, ferronickel and nickel alloys — Determination of sulfur content — Iodimetric titration method after induction furnace combustion*

4.3.14 Silicon, Si (referee method)

ISO 7530-8:1992, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 8: Determination of silicon content*

4.3.15 Tantalum, Ta (referee method)

ISO 23166:2018, *Nickel alloys — Determination of tantalum — Inductively coupled plasma optical emission spectrometric method*

4.3.16 Titanium, Ti (referee method)

ISO 11433:2020, *Nickel alloys — Determination of titanium content — Diantipyrylmethane molecular absorption method*

4.3.17 Vanadium, V (referee method)

ISO 7530-9:1993, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 9: Determination of vanadium content*

5 Range of application and principle of the methods

5.1 Nickels

5.1.1 Silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc [Ag, Bi, Cd, Co, Cu, Fe, Mn, Pb and Zn]

Document: ISO 6351:1985, *Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method*.

Range of application:

- determination in refined, wrought and cast nickel of:
 - silver contents from 0,000 2 % (by mass) to 0,01 % (by mass);
 - bismuth contents from 0,001 0 % (by mass) to 0,01 % (by mass);
 - cadmium contents from 0,000 2 % (by mass) to 0,002 5 % (by mass);

- cobalt contents from 0,001 0 % (by mass) to 1,00 % (by mass);
- copper contents from 0,000 2 % (by mass) to 1,00 % (by mass);
- iron contents from 0,002 5 % (by mass) to 0,15 % (by mass);
- manganese contents from 0,000 5 % (by mass) to 0,20 % (by mass);
- lead contents from 0,000 5 % (by mass) to 0,01 % (by mass);
- zinc contents from 0,000 2 % (by mass) to 0,015 % (by mass).

NOTE 1 The upper limit for the determination of cobalt and copper can be raised to 2 % (by mass) by a minor modification to the method.

NOTE 2 The lower limit for the determination of iron can be extended to less than 0,002 5 % (by mass) provided nickel containing less than 0,000 1 % (by mass) iron is used for preparation of the standard solutions.

Principle of the method:

- a) dissolution of a test portion in nitric acid diluted (1 + 1), evaporation of excess acid and dilution of the solution to a known volume;
- b) aspiration of the solution into the air-acetylene flame of an atomic absorption spectrometer;
- c) measurement of the absorption of the resonance line energy from the spectrum of each element and comparison with that of calibration solutions of the same element in a matched nickel matrix.

5.1.2 Silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium [Ag, As, Bi, Cd, Pb, Sb, Se, Sn, Te and Tl]

Document: ISO 7523:1985, *Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method.*

Range of application:

- determination in refined, wrought and cast nickel of:
 - silver contents from 0,1 µg/g to 10 µg/g;
 - arsenic contents from 1 µg/g to 20 µg/g;
 - bismuth contents from 0,5 µg/g to 15 µg/g;
 - cadmium contents from 0,1 µg/g to 2 µg/g;
 - lead contents from 0,1 µg/g to 10 µg/g;
 - antimony contents from 1 µg/g to 10 µg/g;
 - selenium contents from 1 µg/g to 10 µg/g;
 - tin contents from 1 µg/g to 5 µg/g;
 - tellurium contents from 0,2 µg/g to 10 µg/g;
 - thallium contents from 0,5 µg/g to 10 µg/g.

Principle of the method:

- a) dissolution of a test portion in nitric acid and dilution of the solution to a known volume;
- b) introduction of a known volume of the solution into an electrothermal atomizer of an atomic absorption spectrometer;

- c) measurement of the absorption of the resonance line energy from the spectrum of each element and comparison with that of calibration solutions of the same element in a matched nickel matrix using background correction.

5.1.3 Boron, B

Document: ISO 11436:1993, *Nickel and nickel alloys — Determination of total boron content — Curcumin molecular absorption spectrometric method.*

Range of application:

- determination of total boron contents from 4 µg/g to 240 µg/g in nickel and nickel alloys.

Principle of the method:

- a) dissolution of a test portion in hydrochloric and nitric acids;
- b) decomposition of resistant boron compounds (e.g. boron nitrides) by fuming the sample solution with phosphoric and sulfuric acids at not less than 290 °C for 30 min;
- c) formation of the boron curcumin complex in a buffered acetic acid and sulfuric acid medium;
- d) measurement of the absorbance of the test solution at a wavelength of 543 nm.

5.1.4 Phosphorus, P

Document: ISO 11400:1992, *Nickel, ferronickel and nickel alloys — Determination of phosphorus content — Phosphovanadomolybdate molecular absorption spectrometric method.*

Range of application:

- determination of phosphorus contents from 0,000 5 % (by mass) to 0,05 % (by mass) in nickel, ferronickel and nickel alloys.

NOTE 1 As, Cr, Hf, Nb, Si, Ta, Ti, W interfere, but the interferences can be avoided by complexation or volatilization (for Cr).

NOTE 2 The lowest phosphorus content (0,000 5 %) can only be reached in samples with low contents of the interfering elements.

Principle of the method:

- a) dissolution of a test portion in a mixture of nitric and hydrochloric acids;
- b) fuming with perchloric acid in a PFA or PTFE beaker and removal of chromium as volatile chromylchloride;
- c) complexation of silicon and refractory elements with fluoride ions;
- d) conversion of phosphorus to phosphovanadomolybdic acid in a perchloric and nitric acid solution;
- e) extraction of phosphovanadomolybdic acid into 4-methyl-2-pentanone with citric acid present to complex arsenic.

5.1.5 Sulfur, S

Document: ISO 7527:1985, *Nickel, ferronickel and nickel alloys — Determination of sulfur content — Iodimetric titration method after induction furnace combustion.*

Range of application:

- determination of sulfur contents from:
 - 0,001 % (by mass) to 0,3 % (by mass) in nickel and ferronickel;
 - 0,002 % (by mass) to 0,1 % (by mass) in nickel alloys.

Principle of the method:

- a) combustion of a test portion in a flow of oxygen at a high temperature in a high frequency induction furnace in the presence of fluxes and accelerators;
- b) absorption of the sulfur dioxide formed in an acidified starch-iodide solution and continuous titration with potassium iodate standard volumetric solution.

5.2 Ferronickels

5.2.1 Carbon, C

Document: ISO 7524:2020, *Ferronickels — Determination of carbon content — Infrared absorption method after induction furnace combustion.*

Range of application:

- determination of carbon contents from 0,004 % (by mass) to 2,5 % (by mass).

Principle of the method:

- a) combustion of a test portion in a high-frequency induction furnace at high temperature in a current of pure oxygen, and in the presence of accelerators and fluxes;
- b) transformation of carbon into carbon dioxide and/or carbon monoxide;
- c) measurement by infrared absorption of the carbon dioxide and/or carbon monoxide carried by the current of oxygen.

5.2.2 Chromium, cobalt, copper, manganese and phosphorus [Cr, Co, Cu, Mn and P]

Document: ISO 23156:2021, *Ferronickels — Determination of phosphorus, manganese, chromium, copper and cobalt contents — Inductively coupled plasma optical emission spectrometric method.*

Range of application:

- determination in ferronickels of:
 - cobalt contents from 0,24 % (by mass) to 1,4 % (by mass);
 - chromium contents from 0,076 % (by mass) to 1,86 % (by mass);
 - copper contents from 0,02 % (by mass) to 0,07 % (by mass);
 - manganese contents from 0,02 % (by mass) to 1,0 % (by mass);
 - phosphorus contents from 0,009 % (by mass) to 0,045 % (by mass).

Principle of the method:

- a) dissolution of a test portion with nitric, hydrochloric and hydrofluoric acids;
- b) addition of perchloric acid to remove fluorine and silicon;
- c) addition of nitric and hydrochloric acids to dissolve the salts;

- d) after suitable dilution and, if necessary, addition of an internal reference element, nebulization of the solution into an inductively coupled plasma atomic emission spectrometer and measurement of the intensity of the emitted light.

5.2.3 Cobalt, Co

Document: ISO 7520:1985, *Ferronickel — Determination of cobalt content — Flame atomic absorption spectrometric method*.

Range of application:

- determination of cobalt contents from 0,025 % (by mass) to 2,5 % (by mass).

Principle of the method:

- a) dissolution of a test portion in a mixture of nitric and hydrochloric acids;
b) precipitation of silica by dehydration in perchloric acid;
c) removal of silica by filtration;
d) addition of lanthanum for elimination of potential interferences;
e) determination of cobalt by atomic absorption spectrometry in an air-acetylene flame at a wavelength of 240,7 nm.

5.2.4 Nickel, Ni

Document: ISO 6352:1985, *Ferronickel — Determination of nickel content — Dimethylglyoxime gravimetric method*.

Range of application:

- determination of nickel contents from 15 % (by mass) to 60 % (by mass).

Principle of the method:

- a) dissolution of a test portion in nitric acid;
b) precipitation of silica by dehydration in perchloric acid;
c) removal of silica by filtration;
d) precipitation of nickel from a tartro-ammoniacal medium by an ethanolic solution of dimethylglyoxime;
e) second precipitation of nickel and weighing after drying at 150 °C;
f) determination of residual nickel in the filtrates by atomic absorption spectrometry.

5.2.5 Phosphorus, P

Document: ISO 11400:1992, *Nickel, ferronickel and nickel alloys — Determination of phosphorus content — Phosphovanadomolybdate molecular absorption spectrometric method*.

See [5.1.4](#).

5.2.6 Sulfur, S

Document: ISO 7526:2020, *Ferronickels — Determination of sulfur content — Infrared absorption method after induction furnace combustion*.

Range of application:

- determination of sulfur contents from 0,002 % (by mass) to 0,12 % (by mass).

Principle of the method:

- a) combustion of a test portion in a high-frequency induction furnace at high temperature in a current of pure oxygen, and in the presence of accelerators and fluxes;
- b) transformation of sulfur into sulfur dioxide;
- c) measurement by infrared absorption of the sulfur dioxide carried by the current of oxygen.

Document: ISO 7527:1985, *Nickel, ferronickel and nickel alloys — Determination of sulfur content — Iodimetric titration method after induction furnace combustion*

See [5.1.5](#).

5.2.7 Silicon, Si

Document: ISO 8343:1985, *Ferronickel — Determination of silicon content — Gravimetric method*.

Range of application:

- determination of silicon contents from 0,2 % (by mass) to 4,0 % (by mass).

Principle of the method:

- a) dissolution of a test portion in nitric acid and addition of perchloric acid;
- b) formation of insoluble silica by dehydration in perchloric acid, filtration and weighing of the calcined precipitate;
- c) volatilization of the silica with hydrofluoric and sulfuric acids, weighing of the residue, determination of silica by difference and calculation of the silicon content.

5.3 Nickel alloys

5.3.1 Aluminium, Al

Document: ISO 7530-7:1992, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 7: Determination of aluminium content*.

Range of application:

- determination of aluminium contents from 0,2 % (by mass) to 4,0 % (by mass).

Principle of the method:

- a) dissolution of a test portion in acid, filtration and aspiration of the test solution into a nitrous oxide-acetylene flame of an atomic absorption spectrometer;
- b) combustion of the filter from the acid dissolution and volatilization of silica with hydrofluoric acid;
- c) fusion of the residue with potassium pyrosulfate, dissolution of the melt in dilute acid and aspiration of this second solution into a nitrous oxide-acetylene flame of an atomic absorption spectrometer;
- d) measurement of the absorbance of the resonance line energy from the spectrum of aluminium and comparison with that of calibration solutions at a wavelength of 309,3 nm;
- e) addition of the results found in both solutions.

5.3.2 Boron, B

See 5.1.3.

5.3.3 Cobalt, chromium, copper, iron and manganese [Co, Cr, Cu, Fe and Mn]

Document: ISO 7530-1:2015, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 1: Determination of cobalt, chromium, copper, iron and manganese.*

Range of application:

- determination of cobalt, chromium, copper, iron and manganese from 0,01 % (by mass) to 4,0 % (by mass).

Principle of the method:

- dissolution of a test portion in a mixture of nitric and hydrochloric acids, evaporation of acids excess and dissolution of the salts;
- addition of an ionization suppressant, if necessary, and dilution of the solution to a known volume;
- nebulization of the test solution after suitable dilution, if necessary, into an air/acetylene or a nitrous oxide/acetylene flame of an atomic absorption spectrometer;
- measurement of the absorption of the energy of the resonance line from the spectrum of the spectrum of the element being determined and comparison with that of calibration solutions of the same element.

5.3.4 Chromium, Cr

Document: ISO 7529:2017, *Nickel alloys — Determination of chromium content — Potentiometric titration method with ammonium iron(II) sulfate.*

Range of application:

- determination of chromium from 5 % (by mass) to 22 % (by mass).

Principle of the method:

- dissolution of a test portion in a mixture of nitric and hydrochloric acids, and evaporation to fumes of sulfuric acid;
- dissolution of the salts in water and oxidation of chromium to chromium(VI) with ammonium peroxydisulfate using silver nitrate as a catalyst;
- removal of excess peroxydisulfate by boiling and reduction of manganese(VII) with hydrochloric acid;
- titration of chromium(VI) with ammonium iron(II) sulfate using potentiometric end-point detection.

NOTE 1 The method only applies to nickel alloys which do not contain insoluble carbides and which have a vanadium content less than a mass fraction of 0,2 %.

NOTE 2 Vanadium, which can be present as an impurity in the alloy, gives a positive bias interference. However, at a level of 0,2 % (by mass), this bias is equivalent to 0,068 % (by mass) chromium, which is about half the reproducibility of the method.

5.3.5 Molybdenum, Mo

Document: ISO 11435:2011, *Nickel alloys — Determination of molybdenum content — Inductively coupled plasma atomic emission spectrometric method.*

Range of application:

- determination of molybdenum from 0,05 % (by mass) to 20 % (by mass).

Principle of the method:

- a) dissolution of a test portion in a mixture of hydrochloric, nitric and phosphoric acid, and fuming with a mixture of phosphoric and perchloric acids;
- b) addition of hydrofluoric acid and, if desired, an internal reference element, and dilution of the solution to known volume;
- c) nebulization of the solution into an inductively coupled plasma/atomic emission spectrometer and measurement of the intensity of the emitted light from molybdenum and, if necessary, from the internal reference element, simultaneously.

NOTE The method uses a calibration based on a very close matrix matching of the calibration solutions to the sample and bracketing of the contents between 0,75 and 1,25 of the approximate concentration of molybdenum in the sample to be analysed. The concentration of all elements in the sample has, therefore, to be approximately known. If the concentrations are not known, the sample is analysed by some semi-quantitative method. The advantage with this procedure is that all possible interferences from the matrix are automatically compensated, which results in high accuracy. This is most important for spectral interferences, which can be severe in very highly alloyed metals. All possible interferences are kept at a minimum level. Therefore, it is essential that the spectrometer used meets the performance criteria specified in the method for the selected analytical lines.

5.3.6 Niobium, Nb

Document: ISO 22033:2011, *Nickel alloys — Determination of niobium — Inductively coupled plasma atomic emission spectrometric method.*

Range of application:

- determination of niobium from 0,1 % (by mass) to 10 % (by mass).

Principle of the method:

- a) dissolution of a test portion in a mixture of hydrochloric, nitric and phosphoric acid, and fuming with a mixture of phosphoric and perchloric acids;
- b) addition of hydrofluoric acid and, if necessary, of an internal reference element, and dilution of the solution to known volume;
- c) nebulization of the solution into an inductively coupled plasma atomic emission spectrometer and measurement of the intensity of the emitted light from niobium and, if necessary, from the internal reference element, simultaneously.

NOTE The method uses a calibration based on a very close matrix matching of the calibration solutions to the sample and bracketing of the mass fractions between 0,75 and 1,25 of the approximate concentration of niobium in the sample to be analysed. The concentration of all elements in the sample has, therefore, to be approximately known. If the concentrations are not known, the sample is analysed by some semi-quantitative method. The advantage of this procedure is that all possible interferences from the matrix are automatically compensated, which results in high accuracy. This is most important for spectral interferences, which can be severe in very highly alloyed metals. All possible interferences are kept at a minimum level. Therefore, it is essential that the spectrometer used meets the performance criteria specified in the method for the selected analytical lines.

5.3.7 Nickel, Ni

Document: ISO/TS 18223:2015, *Nickel alloys — Determination of Nickel content — Inductively coupled plasma atomic emission spectrometric method.*

Range of application:

- determination of nickel from 20,0 % (by mass) to 80,0 % (by mass).

NOTE 1 Besides alloys where nickel is the main component regarding its content level, this method can also apply to alloys in which nickel has a content as high as several other elements (Fe, Cr, Co) and in which the “main element” cannot be specified.

Principle of the method:

- a) dissolution of a test portion in nitric and hydrochloric acids;
- b) if necessary, addition of extra hydrofluoric acid and, if desired, addition of an internal reference element (scandium recommended);
- c) dilution of the test solution to a known volume, nebulization of this solution into an inductively coupled plasma atomic emission spectrometer and measurement of the intensity of the emitted light (including, where appropriate, that of the internal reference element).

NOTE 2 The method uses a calibration based on a very close matrix matching of the calibration solutions to the sample composition and a bracketing of the mass fractions between ± 2 % of the approximate content of nickel in the sample to be analysed. The content of all elements in the sample has, therefore, to be approximately known. If the composition is not known, the sample is analysed by some semi-quantitative method. The advantage with this procedure is that all possible matrix interferences are minimized, which results in a higher trueness. This is important for spectral interferences, which can be severe in very highly alloyed matrixes. All possible interferences are kept at a minimum level. Therefore, it is essential that the spectrometer used meets the performance criteria recommended for the selected analytical lines.

5.3.8 Phosphorus, P

Document: ISO 9388:1992, *Nickel alloys — Determination of phosphorus content — Molybdenum blue molecular absorption spectrometric method.*

Range of application:

- determination of phosphorus from 0,001 % (by mass) to 0,025 % (by mass).

NOTE Chromium(III) and silica cause interference which is eliminated.

Principle of the method:

- a) dissolution of a test portion in nitric and hydrochloric acids;
- b) removal of hydrochloric acid by fuming with sulfuric acid;
- c) oxidation of Cr(III) to Cr(VI) with ammonium peroxydisulfate;
- d) precipitation of Fe(III) phosphate and redissolution of the precipitate in a hydrofluoric-nitric acid mixture;
- e) addition of boric, tartaric and sulfamic acids to complex free fluoride, eliminate arsenic interference and remove oxides of nitrogen;
- f) formation and extraction of molybdophosphoric acid into isobutyl alcohol, reduction of the heteropoly acid to phosphovanadomolybdate and back-extraction into an aqueous phase;
- g) measurement of the absorbance of the aqueous solution at 700 nm.

Document: ISO 11400:1992, *Nickel, ferronickel and nickel alloys — Determination of phosphorus content — Molybdenum blue molecular absorption spectrometric method.*

See [5.1.4](#).

5.3.9 Lead, Pb

Document: ISO 11437:2018, *Nickel alloys — Determination of lead — Electrothermal atomic absorption spectrometric method.*

Range of application:

- determination of lead from 1 µg/g to 10 µg/g.

Principle of the method:

- a) dissolution of a test portion in a mixture of nitric acid and hydrofluoric acid, dilution of the test solution to a known volume and transfer of an aliquot to a plastic vial;
- b) addition of a modifier to the aliquot of the test solution and injection of a small volume of this solution into the electrothermal atomizer of an atomic absorption spectrometer;
- c) measurement of the atomic absorption of the 283,3 nm spectral line energy emitted by a lead hollow cathode lamp and comparison with those of the calibration solutions.

5.3.10 Sulfur, S

Document: ISO 7527:1985, *Nickel, ferronickel and nickel alloys — Determination of sulfur content — Iodimetric titration method after induction furnace combustion.*

See [5.1.5](#).

5.3.11 Silicon, Si

Document: ISO 7530-8:1992, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 8: Determination of silicon content.*

Range of application:

- determination of silicon from 0,2 % (by mass) to 1 % (by mass).

Principle of the method:

- a) dissolution of a test portion in acid;
- b) aspiration of the test solution into a nitrous oxide-acetylene flame of an atomic absorption spectrometer;
- c) measurement of the absorbance of the resonance line energy from the spectrum of silicon and comparison with that of calibration solutions at a wavelength of 251,6 nm.

5.3.12 Tantalum, Ta

Document: ISO 23166:2018, *Nickel alloys — Determination of tantalum — Inductively coupled plasma optical emission spectrometric method.*

Range of application:

- determination of tantalum from 0,1 % (by mass) to 5 % (by mass).

Principle of the method:

- a) dissolution of a test portion in a mixture of hydrofluoric, hydrochloric, nitric and phosphoric acid, and fuming after addition of perchloric acid;
- b) addition of hydrofluoric acid and, if desired, of an internal reference element, and dilution of the solution to known volume;
- c) nebulization of the solution into an inductively coupled plasma optical emission spectrometer and measurement of the intensity of the emitted light from tantalum, and, where appropriate, from the internal reference element, simultaneously.

NOTE The method uses a calibration based on a very close matrix matching of the calibration solutions to the sample and bracketing between 0,75 and 1,25 of the approximate content of tantalum in the sample to be analysed. The content of all elements in the sample has, therefore, to be approximately known. If the contents are not known, the sample is analysed by some semi-quantitative method. The advantage of this procedure is that all possible interferences from the matrix are compensated, which results in high accuracy. This is most important for spectral interferences, which can be severe in very highly alloyed matrixes. All possible interferences are kept at a minimum level. Therefore, it is essential that the spectrometer used meets the performance criteria specified in the method for the selected wavelengths.

5.3.13 Titanium, Ti

Document: ISO 11433:2020, *Nickel alloys — Determination of titanium content — Diantipyrylmethane molecular absorption method.*

Range of application:

— determination of titanium from 0,3 % (by mass) to 5,0 % (by mass).

NOTE 1 Evidence exists that extension of this method is possible for titanium contents down to 0,05 % (mass fraction).

NOTE 2 Modifications in the general method allow the determination of titanium in alloys containing tungsten and/or tantalum.

Principle of the method:

- a) dissolution of a test portion with hydrochloric and nitric acids;
- b) elimination of hydrochloric and nitric acids by evaporation to fumes in the presence of sulfuric acid;
- c) formation of a yellow complex with diantipyrylmethane;
- d) spectrophotometric measurement of the absorption of the coloured complex at a wavelength of about 390 nm.

5.3.14 Vanadium, V

Document: ISO 7530:1993, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 9: Determination of vanadium content.*

Range of application:

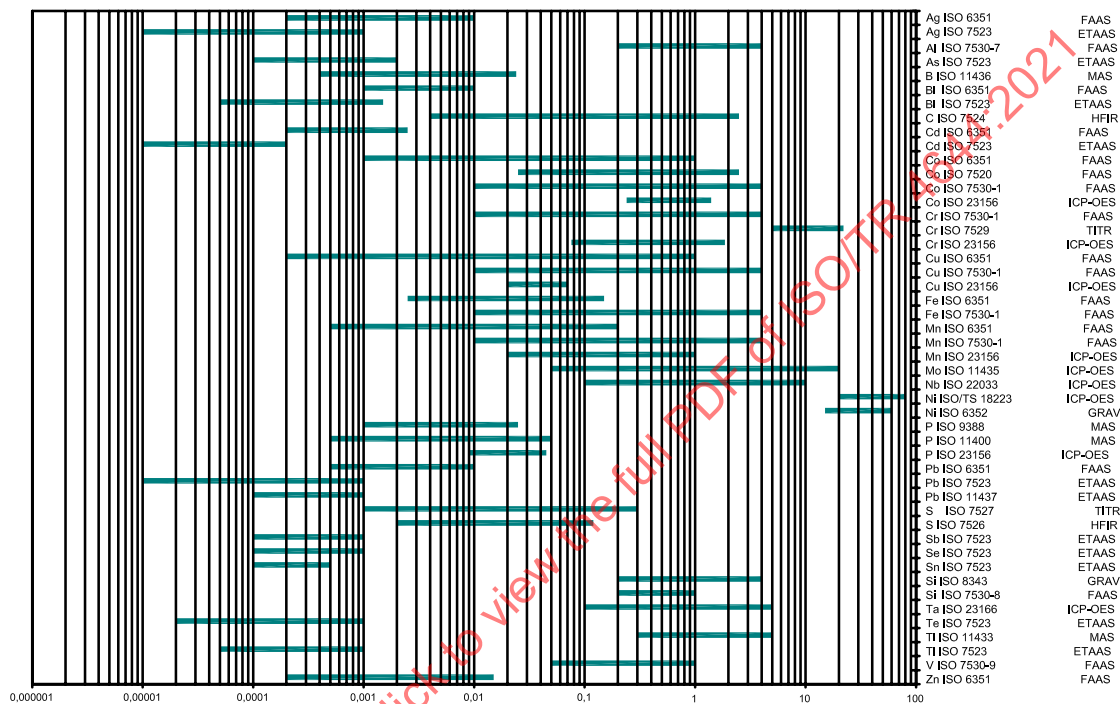
— determination of vanadium from 0,05 % (by mass) to 1,0 % (by mass).

Principle of the method:

- a) dissolution of a test portion in acid;
- b) aspiration of the test solution into a nitrous oxide-acetylene flame of an atomic absorption spectrometer;
- c) measurement of the absorbance of the resonance line energy from the spectrum of vanadium and comparison with that of calibration solutions at a wavelength of 318,4 nm.

Annex A
(informative)

Graphical representation of the scope of methods described in this document



Key

- X range of application (%)
- Y elements and methods

Figure A.1 — Graphical representation of the fields of application of the methods available for the three kinds of matrixes

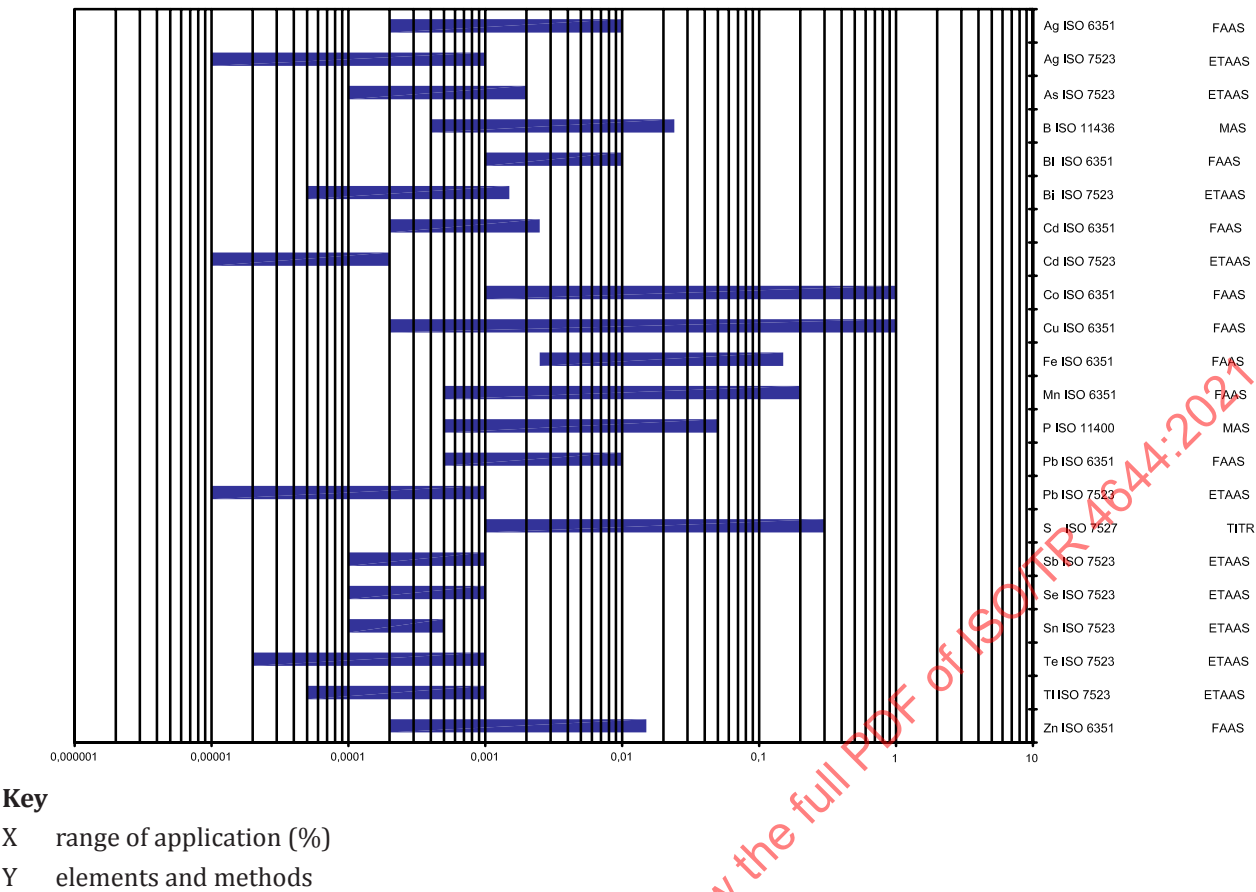


Figure A.2 — Graphical representation of the fields of application of the methods available for nickels